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IS 13146 (1991): Allethrin, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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भारतीय मानक

एलेथ्रीन, तकनीकी — विशिष्टि

Indian Standard

ALLETHRIN, TECHNICAL — SPECIFICATION

UDC 632·951 ALLETHRIN

© BIS 1991

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1991 **Price Group 2**

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Allethrin, technical is employed in the preparation of formulations meant for control of mosquitoes.

Allethrin is the accepted common name by the International Organization for Standardization (ISO) for (*RS*)-3-allyl-2-methyl-4-oxocyclopent-2-enyl (1 *RS*, 2 *RS*; 1 *RS*, 2 *SR*)-2, 2-dimethyl-3-(2-methylprop-1-enyl) = cyclopropanecarboxylate. Its empirical and structural formulae and molecular mass are given below:

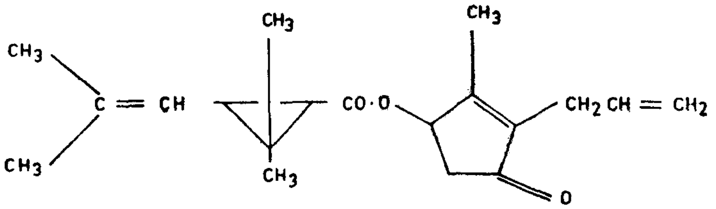
Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{19}H_{26}O_3$		302·4

Fig. 1 Structural Formula

Allethrin, technical generally contains 90 percent (*m/m*) allethrin, out of which minimum 95 percent (*m/m*) is *d*-isomer having a *trans* : *cis* ratio of 3 : 1.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act, 1968* and the Rules framed thereunder. However, this standard is subjected to the restrictions imposed under the *Act* and Rules wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALLETHRIN, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for allethrin, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Water for general laboratory use (*second revision*)
6940 : 1982	Methods of test for pesticides and their formulation (*first revision*)
8190 (Part 2) : 1988	Requirements for packing of pesticides : Part 2 Liquid pesticides (*second revision*)
10946 : 1984	Methods of sampling for technical grade pesticides

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a clear, pale yellowish to brown viscous liquid. It shall be free from extraneous impurities or additives.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed according to the general requirements prescribed in IS 8190 (Part 2) : 1988.

5 MARKING

The containers shall bear legibly and indelibly the following information in addition to any other information as is necessary under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;
b) Indication of the source of manufacture;
c) Date of manufacture and date of expiry;
d) Batch number;
e) *d*-allethrin content, percent (*m/m*);
f) Net mass of the contents; and
g) The minimum cautionary notice as worded in the *Insecticides Act*, 1968 and Rules framed thereunder.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Allethrin, Technical

(*Clauses* 3.2 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Tests, Ref to	
			Annex of This Standard	Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	*d*-Allethrin content, percent by mass, *Min*	85·5	A	—
ii)	Ratio of *trans* and *cis* isomers	*trans* : *cis* = 3 : 1	B	—
iii)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9
iv)	Relative density at 27°C	1·000 to 1·020	—	5
v)	Acidity (as H_2SO_4), percent by mass, *Max*	1·0	—	11.3.2

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF *d*-ALLETHRIN CONTENT

A-1 PRINCIPLE

A-1.1 *d*-allethrin is dissolved in toluene and then gas chromatographically estimated using a Flame Ionization Detecor (FID).

A-2 APPARATUS

A-2.1 Microlitre Syringe

10-µl capacity.

A-2.2 Gas Chromatograph (**GLC**)

Equipped with a FID and may be coupled to a printer-plotter *cum* integrator. The operative parameters are suggested below, which can be changed, in any other GLC equipment employed, provided standardization is done:

Column — Glass, 50 cm length and 3 mm ID packed with 5 percent *OV* 101 on Chromosorb *WHP* (80-100 mesh)

Temperatures

Column oven	185°C
Injection port	230°C
Detector	250°C

Gases and Corresponding Flow Rates

Nitrogen (carrier)	25 ml/min
Hydrogen	25 ml/min
Air	100 ml/min

A-3 REAGENTS

A-3.1 Toluene

AR grade

A-3.2 Internal Standard Solution

Dibutyl phthalate, AR grade

A-3.3 *d*-allethrin Reference Standard

Of known purity

A-4 PROCEDURE

A-4.1 Preparation of Internal Standard Solution

Weigh accurately 1·0 g of dibutyl phthalate in a 100-ml volumetric flask and make up the volume with toluene. This will give a solution containing 10 mg/ml of dibutyl phthalate.

A-4.2 Preparation of Standard Solution

Weigh accurately 200 mg of *d*-allethrin reference standard in a 100-ml volumetric flask, add about 25-35 ml of toluene and shake well. Add 10 ml of the internal standard solution and finally make up the volume with toluene.

A-4.3 Preparation of Sample Solution

Weigh accurately a quantity of the sample equivalent to 200 mg of *d*-allethrin in a 100 ml volumetric flask, add about 25-35 ml of toluene and shake well. Add 10 ml of the internal standard solution finally make up the volume with toluene and mix the contents thoroughly.

A-4.4 Estimation

Inject a known volume of the standard solution and the sample solution with the help of a microlitre syringe. Measure the peak heights/areas of the internal standard, standard and sample.

A-5 CALCULATION

d-allethrin content, percent by mass

$$= \frac{M_1 \times A_1 \times A_3 \times P}{M_2 \times A_2 \times A_4}$$

where

M_1 = Mass in g of standard *d*-allethrin;

A_1 = Peak height/area of internal standard in the reference standard *d*-allethrin solution injected;

A_3 = Peak height/area of *d*-allethrin in the sample solution injected;

P = Percent purity of *d*-allethrin reference standard;

M_2 = Mass in g of sample taken for the test;

A_2 = Peak height/area of *d*-allethrin in the reference standard *d*-allethrin solution injected; and

A_4 = Peak height/area of internal standard in the sample solution injected.

ANNEX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF *TRANS* AND *CIS* RATIO IN ALLETHRIN, TECHNICAL

B-1 APPARATUS

B-1.1 Gas Chromatograph (GLC)

Equipped with a hydrogen flame ionization detector and an integrator-*cum*-printer-plotter. The operative parameters are suggested below, which can be changed, in any other GLC equipment employed, provided standardization is done:

Column — Glass, 30 cm length and 2·5 mm ID, coated with 20 percent (*m*/*v*) solution of *DCQF*-1 in acetone.

Temperatures

Column oven	150°C
Injection port	200°C
Detector	200°C

Carrier Gas and Flow Rate

Nitrogen	100 ml/min

B-1.2 Microlitre Syringe

10-μl capacity.

B-2 REAGENTS

B-2.1 Methanol

AR grade

B-2.2 Methanol

50 percent (*v*/*v*) solution.

B-2.3 Methanolic Potassium Hydroxide Solution

1 N, prepared using methanol (**B-2.2**).

B-2.4 Chloroform

AR grade

B-2.5 Sodium Sulphate

Anhydrous, AR grade

B-2.6 Dilute Hydrochloric Acid

1 : 1 (*v*/*v*).

B-2.7 Toluene

AR grade.

B-2.8 Toluene Solution

2 percent (*m*/*v*).

B-2.9 Thionylchloride Solution

8 percent (*m*/*v*) in toluene.

B-2.10 *d*-2-Octanol Solution

20 percent (*m*/*v*) in toluene using *d*-2-octanol of known purity.

B-3 PROCEDURE

B-3.1 Weigh accurately about 0·015 g of the sample and dissolve it in 5 ml methanol (**B-2.1**) and add 20 ml of methanolic potassium hydrooxide solution. Heat the mixture on a water-bath under reflux for 30 minutes. Cool the solution, add 20 ml of water and wash with three 10 ml portions of chloroform. Acidify the solution with 10 ml of dilute hyrochloric acid, and extract swirling vigorously with three 10 ml portions of chloroform.

B-3.2 Combine all the chloroform extracts, dry over 5 g sodium sulphate, filter the solution and wash the residue with 10 ml chloroform. Combine the filtrate, the washings and distill off chloroform in vacuum. Add 0·25 ml toluene solution, 0·25 ml thionylchloride solution and 0·25 ml *d*-2-octanol solution, in that order to the residue. Heat the mixture on a water-bath for 2 hours.

B-3.3 Inject 2·0 μl of the solution into the gas chromatograph and with the help of an integrator, calculate peaks areas of isomers. There will be four major peaks. The peaks of *d-cis*, *l-cis*, *d-trans* and *l-trans* isomers will appear at the position of retention time ratio 1·0, 1·07, 1·14 and 1·23 respectively.

B-4 CALCULATION

B-4.1 *cis*-isomers, percent by mass, (*d-cis* + *l-cis* isomers) in the allethrin, technical (*A*)

$$= \frac{\text{Total area of the first two major peaks}}{\text{Total area of all the four major peaks}} \times 100$$

B-4.2 *trans*-isomers, percent by mass (*d-cis* + *l-cis* isomers) in the allethrin technical samples (*B*)

$$= \frac{\text{Total area of the third and fourth major peaks}}{\text{Total area of all the four major peaks}} \times 100$$

B-4.3 Ratio of *trans* : *cis* isomers $= \frac{B}{A}$

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Doc : No. FADC 1 (4176)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at New India Printing Press, Khurja, India